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FLATT
2728-2732

L-2732-M1

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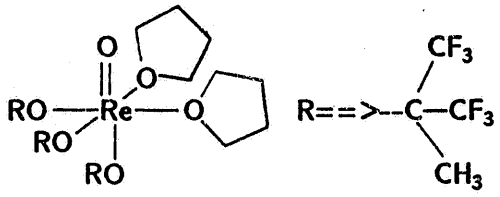
Notebook references
E72625-52
E65724-94

TO: Jerry Feldman, CRD, E328/317, x54669

FROM: J. Calabrese, CRD, E228/320B, x3952

X-RAY CRYSTAL STRUCTURE ANALYSIS 92078
$Re(O)[CMe(CF_3)_2]_3(THF)_2$



CRYSTAL DATA: $ReF_{18}O_6C_{20}H_{25}$, from hexanes/THF, blue, wedge, ~0.35 x 0.26 x 0.49mm, monoclinic, $P2_1/n$ (No. 14), a = 10.010(3), b = 29.247(6), c = 10.800(3)Å, β = 117.09(1)°, from 25 reflections, T = -70°C, V = 2815.0Å^3, Z = 4, FW = 889.64, Dc = 2.099g/cc, μ(Mo) = 45.25cm^{-1}.

DATA COLLECTION AND TREATMENT: Enraf-Nonius CAD4 diffractometer, MoKα radiation, 6891 data collected, 1.4° < 2θ < 55.0°, maximum h,k,l = 12 37 14, data octants = +++, -++, ω scan method, scan width = 1.20-2.20°ω, scan speed = 1.70-5.00°/min, typical half-height peak width = 0.15°ω, 2 standards collected 49 times, adjusted for a 4% decrease in intensity, 13.2% variation in azimuthal scan, corrected for absorption (azimuthal), range of transmission factors = 0.24-0.26, 227 duplicates, 1.6% R-merge, 4447 unique reflections with I> 3.0σ(I).

SOLUTION AND REFINEMENT: Structure solved by automated Patterson analysis (PHASE), [The asymmetric unit consists of one molecule in a general position] refinement by full-matrix least squares on F, scattering factors from Int. Tables for X-ray Crystallography, Vol IV, including anomalous terms for Re, weights $\propto [\sigma^2(I)+0.00091^2]^{-1/2}$, refined anisotropic: all non-hydrogen atoms, fixed atoms: H , 406 parameters, data/parameter ratio = 10.95, final R = 0.035, Rw = 0.038, error of fit = 1.45, max Δ/σ = 0.44, largest residual density = 0.88e/Å^3, (background)

RESULTS: This analysis determines the structure of tris[tert-bis(trifluoromethyl)-methylbutoxy]-rhenium(V) oxo bis(tetrahydrofuran). This complex serves as a precursor for potential olefin metathesis catalysts. The rhenium atom is approximately octahedrally coordinated to the six oxygen atoms. The butoxy ligands are arranged in a facial arrangement. The environment around the rhenium center is sterically crowded as evidenced by one long Rh-THF oxygen bond (RE1-O6 2.236Å) and by the distortion from octahedral symmetry (O1-RE1-O2 162°).

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TABLE I. Fractional Coordinates (X10000) and Isotropic Thermal Parameters

ATOM	X	Y	Z	BISO
Re(1)	5939.3(3)	6073.2(1)	5629.6(3)	1.7(1)'
F(21)	6615(11)	7498(2)	5705(9)	10.3(5)'
F(22)	7260(8)	7726(2)	4161(8)	8.2(4)'
F(23)	5109(7)	7461(2)	3590(10)	10.6(4)'
F(24)	5507(10)	6794(4)	2109(6)	13.1(4)'
F(25)	7529(11)	6408(2)	3030(10)	11.6(6)'
F(26)	7687(10)	7108(2)	2643(7)	10.0(4)'
F(31)	3921(6)	6021(2)	7622(5)	5.3(2)'
F(32)	5930(6)	6042(2)	9523(6)	6.2(2)'
F(33)	4169(6)	6510(2)	9181(5)	5.7(2)'
F(34)	7789(5)	6733(2)	10000(5)	5.2(2)'
F(35)	6052(6)	7230(2)	9577(5)	5.0(2)'
F(36)	7284(6)	7235(2)	8408(5)	5.3(2)'
F(41)	11019(5)	6248(2)	7720(6)	5.6(2)'
F(42)	11378(6)	6012(2)	9749(5)	6.6(2)'
F(43)	9710(6)	6518(2)	8661(5)	5.1(2)'
F(44)	8871(8)	5011(2)	7050(6)	7.8(3)'
F(45)	10373(6)	5419(2)	6592(5)	6.1(2)'
F(46)	11042(6)	5188(2)	8677(6)	7.1(2)'
O(1)	5277(5)	5570(1)	5864(5)	2.6(2)'
O(2)	6215(5)	6623(1)	4761(4)	2.3(1)'
O(3)	6357(5)	6387(2)	7367(4)	2.2(1)'
O(4)	8083(5)	5919(1)	6474(4)	2.0(1)'
O(5)	5810(5)	5757(1)	3749(4)	2.5(1)'
O(6)	3522(5)	6260(2)	4384(5)	2.6(1)'
C(21)	7089(8)	6923(2)	4456(7)	2.6(2)'
C(22)	8714(11)	6914(4)	5499(11)	6.4(4)'
C(23)	6489(15)	7402(3)	4414(14)	6.2(5)'
C(24)	6959(15)	6797(5)	3037(12)	6.9(5)'
C(31)	5526(8)	6639(3)	7860(7)	2.8(2)'
C(32)	4264(9)	6935(3)	6828(8)	3.8(3)'
C(33)	4894(9)	6307(3)	8563(9)	3.9(3)'
C(34)	6683(9)	6962(3)	8989(9)	3.7(3)'
C(41)	9126(8)	5770(3)	7800(7)	2.9(2)'
C(42)	8498(9)	5621(3)	8799(8)	4.1(3)'
C(43)	10299(9)	6144(3)	8468(8)	3.9(3)'
C(44)	9872(11)	5353(3)	7512(10)	5.2(3)'
C(52)	6916(9)	5415(3)	3831(8)	3.7(3)'
C(53)	6428(10)	5281(3)	2327(8)	4.3(3)'
C(54)	4774(10)	5334(3)	1653(9)	4.5(3)'
C(55)	4413(9)	5631(4)	2568(9)	5.5(3)'
C(62)	2906(8)	6628(2)	3350(8)	3.4(2)'
C(63)	1239(10)	6596(3)	2820(10)	5.2(3)'
C(64)	963(9)	6104(3)	2983(11)	5.4(3)'
C(65)	2285(8)	5974(3)	4335(8)	3.9(3)'
H(22)	9258	7126	5238	5.5
H(22')	9110	6615	5536	5.5
H(22")	8816	6993	6392	5.5
H(32)	3787	7090	7293	5.5
H(32')	3552	6748	6113	5.5
H(32")	4662	7153	6433	5.5
H(42)	9299	5529	9662	5.5
H(42')	7826	5372	8405	5.5
H(42")	7979	5870	8950	5.5
H(52)	6853	5157	4334	5.5

TABLE I. Fractional Coordinates (X10000) and Isotropic Thermal Parameters

H(52')	7882	5552	4205	5.5
H(53)	6674	4970	2285	5.5
H(53')	6843	5489	1920	5.5
H(54)	4320	5043	1575	5.5
H(54')	4450	5477	773	5.5
H(55)	3918	5899	2074	5.5
H(55')	3804	5468	2881	5.5
H(62)	3146	6571	2608	5.5
H(62')	3252	6914	3793	5.5
H(63)	749	6676	1863	5.5
H(63')	950	6777	3390	5.5
H(64)	987	5932	2246	5.5
H(64')	66	6074	3074	5.5
H(65)	2085	6053	5087	5.5
H(65')	2520	5661	4308	5.5

TABLE II. Anisotropic Thermal Parameters (Angs X 1000)
exp[-19.739(U11hha*a*...+2(U12hka*b*...))]

ATOM	U11	U22	U33	U12	U13	U23
Re(1)	19.5(1)	20.0(1)	22.4(1)	0.4(1)	6.4(1)	0.6(1)
F(21)	237(10)	47(4)	191(8)	-13(5)	169(8)	-25(4)
F(22)	126(5)	38(3)	193(7)	0(3)	110(5)	31(4)
F(23)	65(4)	65(4)	266(11)	31(3)	69(6)	87(6)
F(24)	163(8)	262(12)	38(4)	-113(9)	17(5)	-4(5)
F(25)	252(11)	69(5)	254(11)	-54(6)	232(10)	-64(6)
F(26)	228(9)	111(5)	101(5)	-86(6)	128(6)	-34(4)
F(31)	72(3)	72(4)	75(4)	-33(3)	49(3)	-26(3)
F(32)	72(4)	104(5)	72(4)	9(4)	44(3)	38(4)
F(33)	67(3)	101(4)	74(4)	-20(3)	55(3)	-27(3)
F(34)	50(3)	89(4)	38(3)	8(3)	1(2)	-26(3)
F(35)	64(3)	68(3)	59(3)	0(3)	28(3)	-33(3)
F(36)	78(4)	57(3)	79(4)	-32(3)	46(3)	-28(3)
F(41)	37(3)	79(4)	91(4)	-15(3)	25(3)	4(3)
F(42)	56(3)	88(4)	57(3)	-6(3)	-19(3)	9(3)
F(43)	66(3)	52(3)	64(3)	-15(3)	20(3)	-17(3)
F(44)	115(5)	33(3)	99(5)	5(3)	5(4)	-1(3)
F(45)	69(4)	104(5)	53(3)	47(3)	21(3)	1(3)
F(46)	83(4)	88(4)	66(4)	55(4)	3(3)	17(3)
O(1)	30(2)	24(2)	39(3)	-6(2)	11(2)	2(2)
O(2)	32(2)	25(2)	31(2)	-1(2)	16(2)	4(2)
O(3)	25(2)	33(3)	25(2)	-1(2)	11(2)	-4(2)
O(4)	23(2)	23(2)	27(2)	0(2)	8(2)	4(2)
O(5)	32(3)	26(2)	30(2)	2(2)	9(2)	-7(2)
O(6)	23(2)	33(2)	38(3)	0(2)	8(2)	0(2)
C(21)	42(4)	25(4)	33(4)	-4(3)	20(3)	7(3)
C(22)	51(6)	86(8)	75(7)	-36(5)	1(5)	34(6)
C(23)	113(10)	43(6)	115(10)	2(6)	82(9)	16(6)
C(24)	108(10)	108(10)	67(7)	-56(8)	58(7)	-10(7)
C(31)	33(4)	46(4)	28(4)	-4(3)	14(3)	-12(3)
C(32)	39(4)	50(5)	48(5)	14(4)	13(4)	-11(4)
C(33)	43(5)	69(6)	47(5)	-10(4)	31(4)	-13(4)
C(34)	46(5)	51(5)	49(5)	-6(4)	28(4)	-22(4)
C(41)	27(4)	38(4)	31(4)	5(3)	3(3)	4(3)
C(42)	48(5)	52(5)	40(4)	1(4)	5(4)	22(4)
C(43)	41(4)	46(5)	42(4)	2(4)	3(4)	7(4)
C(44)	58(6)	49(6)	56(6)	27(5)	-3(5)	4(5)
C(52)	47(5)	44(5)	47(5)	20(4)	18(4)	-7(4)

TABLE II. Anisotropic Thermal Parameters (Angs X 1000) (continued)

C(53)	52(5)	62(6)	46(5)	11(4)	18(4)	-14(4)
C(54)	56(6)	72(6)	44(5)	-11(5)	23(4)	-20(5)
C(55)	38(5)	96(8)	50(5)	9(5)	-2(4)	-44(5)
C(62)	35(4)	31(4)	43(4)	10(3)	1(3)	7(3)
C(63)	37(5)	63(6)	74(6)	12(4)	3(4)	6(5)
C(64)	28(4)	61(6)	87(7)	-3(5)	1(4)	-7(6)
C(65)	30(4)	60(6)	47(5)	-10(4)	8(3)	-8(4)

TABLE III. Interatomic Distances (A)

Re(1)-O(1)	1.681 (4)
Re(1)-O(2)	1.943 (4)
Re(1)-O(3)	1.956 (4)
Re(1)-O(4)	1.964 (4)
Re(1)-O(5)	2.180 (4)
Re(1)-O(6)	2.236 (4)
F(21)-C(23)	1.370 (13)
F(22)-C(23)	1.326 (10)
F(23)-C(23)	1.269 (13)
F(24)-C(24)	1.338 (14)
F(25)-C(24)	1.275 (15)
F(26)-C(24)	1.350 (11)
F(31)-C(33)	1.333 (9)
F(32)-C(33)	1.331 (10)
F(33)-C(33)	1.329 (9)
F(34)-C(34)	1.330 (9)
F(35)-C(34)	1.334 (8)
F(36)-C(34)	1.319 (9)
F(41)-C(43)	1.340 (10)
F(42)-C(43)	1.367 (8)
F(43)-C(43)	1.304 (9)
F(44)-C(44)	1.341 (11)
F(45)-C(44)	1.315 (12)
F(46)-C(44)	1.358 (9)
O(2)-C(21)	1.381 (7)
O(3)-C(31)	1.386 (8)
O(4)-C(41)	1.403 (7)
O(5)-C(52)	1.465 (8)
O(5)-C(55)	1.447 (8)
O(6)-C(62)	1.469 (8)
O(6)-C(65)	1.474 (9)
C(21)-C(22)	1.498 (11)
C(21)-C(23)	1.518 (12)
C(21)-C(24)	1.523 (12)
C(31)-C(32)	1.518 (10)
C(31)-C(33)	1.536 (11)
C(31)-C(34)	1.561 (10)
C(41)-C(42)	1.538 (11)
C(41)-C(43)	1.524 (10)
C(41)-C(44)	1.534 (11)
C(52)-C(53)	1.519 (10)
C(53)-C(54)	1.482 (12)
C(54)-C(55)	1.480 (11)
C(62)-C(63)	1.500 (11)
C(63)-C(64)	1.491 (13)
C(64)-C(65)	1.506 (11)

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TABLE IV. Intramolecular Angles (Deg)

O(1)-Re(1)-O(2)	161.8 (2)	F(22)-C(23)-C(21)	113.9 (8)
O(1)-Re(1)-O(3)	101.2 (2)	F(23)-C(23)-C(21)	116 (1)
O(1)-Re(1)-O(4)	98.7 (2)	F(24)-C(24)-C(21)	109 (1)
O(1)-Re(1)-O(5)	84.7 (2)	F(25)-C(24)-C(21)	113 (1)
O(1)-Re(1)-O(6)	84.9 (2)	F(26)-C(24)-C(21)	110.1 (9)
O(2)-Re(1)-O(3)	93.4 (2)	F(31)-C(33)-C(31)	110.0 (6)
O(2)-Re(1)-O(4)	92.8 (2)	F(32)-C(33)-C(31)	113.7 (6)
O(2)-Re(1)-O(5)	82.1 (2)	F(33)-C(33)-C(31)	114.0 (8)
O(2)-Re(1)-O(6)	82.0 (2)	F(34)-C(34)-C(31)	112.3 (7)
O(3)-Re(1)-O(4)	87.2 (2)	F(35)-C(34)-C(31)	112.5 (6)
O(3)-Re(1)-O(5)	171.3 (2)	F(36)-C(34)-C(31)	109.4 (6)
O(3)-Re(1)-O(6)	99.3 (2)	F(41)-C(43)-C(41)	112.8 (7)
O(4)-Re(1)-O(5)	85.6 (2)	F(42)-C(43)-C(41)	111.1 (7)
O(4)-Re(1)-O(6)	171.8 (2)	F(43)-C(43)-C(41)	111.8 (7)
O(5)-Re(1)-O(6)	87.4 (2)	F(44)-C(44)-C(41)	108.5 (8)
Re(1)-O(2)-C(21)	152.3 (4)	F(45)-C(44)-C(41)	115.0 (8)
Re(1)-O(3)-C(31)	135.6 (4)	F(46)-C(44)-C(41)	112.8 (7)
Re(1)-O(4)-C(41)	133.9 (4)	O(2)-C(21)-C(22)	113.5 (6)
Re(1)-O(5)-C(52)	120.7 (4)	O(2)-C(21)-C(23)	107.9 (6)
Re(1)-O(5)-C(55)	123.6 (4)	O(2)-C(21)-C(24)	108.3 (6)
Re(1)-O(6)-C(62)	126.9 (4)	O(3)-C(31)-C(32)	117.7 (6)
Re(1)-O(6)-C(65)	123.0 (4)	O(3)-C(31)-C(33)	108.0 (6)
C(52)-O(5)-C(55)	106.2 (5)	O(3)-C(31)-C(34)	105.1 (6)
C(62)-O(6)-C(65)	109.4 (5)	O(4)-C(41)-C(42)	116.8 (6)
F(21)-C(23)-F(22)	106 (1)	O(4)-C(41)-C(43)	108.3 (6)
F(21)-C(23)-F(23)	105 (1)	O(4)-C(41)-C(44)	104.2 (6)
F(22)-C(23)-F(23)	108 (1)	O(5)-C(52)-C(53)	104.3 (6)
F(24)-C(24)-F(25)	109 (1)	O(5)-C(55)-C(54)	107.8 (7)
F(24)-C(24)-F(26)	109 (1)	O(6)-C(62)-C(63)	104.7 (7)
F(25)-C(24)-F(26)	107 (1)	O(6)-C(65)-C(64)	104.2 (7)
F(31)-C(33)-F(32)	105.6 (8)	C(22)-C(21)-C(23)	108.5 (8)
F(31)-C(33)-F(33)	106.7 (6)	C(22)-C(21)-C(24)	108.6 (8)
F(32)-C(33)-F(33)	106.2 (7)	C(23)-C(21)-C(24)	110.0 (8)
F(34)-C(34)-F(35)	107.7 (6)	C(32)-C(31)-C(33)	109.3 (6)
F(34)-C(34)-F(36)	107.8 (7)	C(32)-C(31)-C(34)	107.5 (6)
F(35)-C(34)-F(36)	106.8 (7)	C(33)-C(31)-C(34)	108.9 (6)
F(41)-C(43)-F(42)	106.3 (7)	C(42)-C(41)-C(43)	110.1 (6)
F(41)-C(43)-F(43)	108.1 (7)	C(42)-C(41)-C(44)	108.0 (7)
F(42)-C(43)-F(43)	106.3 (7)	C(43)-C(41)-C(44)	109.0 (7)
F(44)-C(44)-F(45)	107.9 (8)	C(52)-C(53)-C(54)	104.0 (7)
F(44)-C(44)-F(46)	105.3 (8)	C(53)-C(54)-C(55)	107.0 (7)
F(45)-C(44)-F(46)	106.7 (8)	C(62)-C(63)-C(64)	104.4 (7)
F(21)-C(23)-C(21)	107.8 (9)	C(63)-C(64)-C(65)	103.3 (7)

TABLE V. Intramolecular Non-Bonding Distances (A)

Re(1)...F(21)	4.216 (6)	F(43)...O(3)	3.015 (7)
Re(1)...F(24)	4.195 (7)	F(44)...O(1)	3.613 (8)
Re(1)...F(25)	3.929 (6)	F(21)...C(32)	3.510 (11)
Re(1)...F(31)	3.562 (5)	F(23)...C(62)	3.217 (10)
Re(1)...F(32)	4.210 (5)	F(24)...C(55)	3.675 (15)
Re(1)...F(36)	4.323 (5)	F(24)...C(62)	3.463 (12)
Re(1)...F(43)	3.925 (5)	F(25)...C(52)	3.167 (11)
Re(1)...F(44)	4.064 (6)	F(25)...C(53)	3.448 (11)
F(21)...F(36)	2.789 (10)	F(25)...C(55)	3.703 (12)
F(22)...F(24)	3.445 (11)	F(31)...C(65)	3.164 (9)
F(22)...F(26)	2.602 (9)	F(32)...C(42)	3.251 (11)
F(23)...F(24)	2.665 (14)	F(34)...C(42)	3.687 (10)
F(23)...F(26)	3.343 (11)	F(41)...C(22)	3.136 (11)
F(32)...F(34)	2.634 (8)	F(43)...C(22)	3.301 (11)
F(32)...F(35)	3.476 (8)	F(43)...C(34)	3.461 (10)
F(33)...F(34)	3.379 (7)	F(44)...C(52)	3.335 (10)
F(33)...F(35)	2.726 (7)	F(45)...C(52)	3.387 (9)
F(34)...F(43)	2.952 (7)	O(1)...C(33)	3.783 (9)
F(36)...F(43)	3.126 (8)	O(1)...C(42)	3.340 (9)
F(41)...F(45)	2.656 (8)	O(2)...C(32)	3.688 (9)
F(41)...F(46)	3.264 (8)	O(3)...C(42)	2.998 (9)
F(42)...F(45)	3.538 (8)	O(3)...C(43)	3.636 (9)
F(42)...F(46)	2.628 (8)	O(4)...C(22)	3.253 (10)
F(21)...O(3)	3.776 (8)	O(5)...C(24)	3.460 (12)
F(24)...O(5)	3.456 (11)	O(6)...C(32)	3.104 (9)
F(25)...O(4)	3.782 (10)	C(22)...C(43)	3.639 (12)
F(25)...O(5)	2.899 (8)	C(32)...C(62)	3.481 (11)
F(31)...O(1)	3.088 (7)	C(44)...C(52)	3.726 (11)
F(31)...O(6)	3.406 (7)	C(55)...C(62)	3.555 (12)
F(32)...O(1)	3.950 (7)	C(55)...C(65)	3.590 (12)

TABLE VI. Intermolecular Distances (A)

F(22)...F(33)a	2.934 (8)
F(22)...F(41)b	3.350 (8)
F(22)...F(43)b	3.232 (8)
F(23)...F(36)b	2.883 (8)
F(23)...F(43)b	3.019 (8)
F(24)...F(33)c	2.936 (8)
F(24)...F(35)c	3.280 (9)
F(26)...F(34)c	3.104 (8)
F(26)...F(35)c	2.973 (9)
F(31)...F(41)d	3.029 (7)
F(33)...F(41)d	2.914 (7)
F(36)...C(62)e	3.389 (9)
F(42)...C(55)f	3.363 (10)
F(44)...C(64)g	3.266 (11)
F(44)...C(65)g	3.209 (10)

Symmetry Operation Codes

a	1/2+X,3/2-Y,-1/2+Z
b	-1/2+X,3/2-Y,-1/2+Z
c	X,Y,-1+Z
d	-1+X,Y,Z
e	1/2+X,3/2-Y,1/2+Z
f	1+X,Y,1+Z
g	1-X,1-Y,1-Z

L-2732-M7

E. I. DU PONT DE NEMOURS & CO., INC.
Central Research & Development Dept.
Experimental Station

5-OCT-92

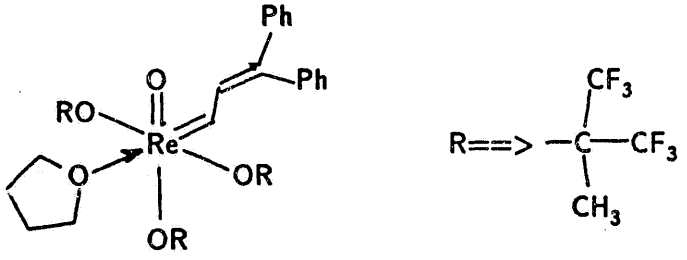
Notebook references
E76053-88
E65724-123

TO: B. Flatt, J. Feldman, CRD, E328/317,x56449

FROM: N. Jones, CRD, E228/316, x9674

X-RAY CRYSTAL STRUCTURE ANALYSIS 92176

$ReC_{31}H_{29}O_5F_{18}$



CRYSTAL DATA: $ReF_{18}O_5C_{31}H_{29}$, from pet ether ($-_{40}$C), orange-brown, parallelopiped, ~0.36 x 0.16 x 0.52mm, triclinic, $P\bar{1}$ (No. 2), a = 10.459(3), b = 10.913(3), c = 21.308(6)Å, α = 91.16(3), β = 102.05(2), γ = 117.98(2)°, from 25 reflections, T = -65° C, V = 2080.7Å^3, Z = 2, FW = 1009.80, Dc = 1.612g/cc, μ(Mo) = 30.69cm^{-1},

DATA COLLECTION AND TREATMENT: Enraf-Nonius CAD4 diffractometer, MoKα radiation, 6707 data collected, 2.0° < 2θ < 48.0°, maximum h,k,l = 11 12 24, data octants = +++, +-+, -++, --+, ω scan method, scan width = 1.20-2.20° ω, scan speed = 1.70-4.00°/min, typical half-height peak width = 0.20° ω, 2 standards collected 54 times, adjusted for a 5% decrease in intensity, 14.4% variation in azimuthal scan, corrected for absorption (azimuthal), range of transmission factors = 0.29-0.34, 162 duplicates, 1.7% R-merge, 4987 unique reflections with I> 3.0σ(I),

SOLUTION AND REFINEMENT: Structure solved by automated Patterson analysis (PHASE), [The asymmetric unit consists of one molecule in a general position. Hydrogen atoms were idealized with C-H=.95Å.], refinement by full-matrix least squares on F, scattering factors from Int. Tables for X-ray Crystallography, Vol IV, including anomalous terms for Re, biweighted unit weights, (excluded 21), refined anisotropic: all non-hydrogen atoms, fixed atoms: H , 496 parameters, data/parameter ratio = 10.01, final R = 0.054, Rw = 0.056, error of fit = 5.40, max Δ/σ = 1.10, [the fluorine atoms on C23 and C24 show high thermal motion and oscillation in the refinement. Attempts to model half-atom disorder were unsuccessful] largest residual density = 2.36e/Å^3, near RE1

RESULTS: This analysis determines the structure of rhenium(VII) oxotris(hexafluoro-t-butoxide)-diphenylvinylcarbene- tetrahydrofuran as part of an investigation of olefin metathesis chemistry. Unlike the previous analysis (92078) of a bis(THF) analog in which the three butoxy ligands are facially coordinated to octahedral Re, here the butoxy groups are cis to each other and lie in the equatorial plane defined by the oxo ligand. The THF and alkylidene ligands occupy the trans-axial positions (Fig. 1). The Re-O distances vary from 1.674 (oxo), 1.954(av-butoxy), to 2.315 (THF).

L-2732-MS

TABLE I. Fractional Coordinates (X10000) and Isotropic Thermal Parameters

ATOM	X	Y	Z	BISO
Re(1)	3494.8(5)	4209.0(4)	2028.7(2)	2.4(1)
F(21)	5336(13)	2593(15)	827(6)	10.7(7)
F(22)	4358(26)	3837(12)	259(7)	17.0(12)
F(23)	3508(30)	1760(15)	-173(7)	21.9(15)
F(24)	1151(21)	296(14)	1073(10)	15.9(10)
F(25)	3609(31)	757(19)	1401(6)	20.5(18)
F(26)	2051(17)	-264(12)	425(6)	12.1(7)
F(31)	6793(8)	6338(8)	3273(3)	5.7(3)
F(32)	7074(9)	7950(8)	2680(4)	6.8(4)
F(33)	8902(8)	7607(9)	3077(4)	7.3(4)
F(34)	7712(10)	7662(10)	1540(5)	8.0(4)
F(35)	9315(9)	7068(11)	1942(5)	8.7(4)
F(36)	7415(10)	5691(11)	1195(4)	8.8(4)
F(41)	-1526(9)	2960(12)	1422(5)	8.1(5)
F(42)	-1284(8)	1269(11)	1050(4)	8.2(4)
F(43)	-2512(8)	936(11)	1769(5)	8.9(4)
F(44)	424(9)	4518(9)	2560(4)	6.2(3)
F(45)	1324(8)	3476(9)	3198(3)	5.8(3)
F(46)	-1058(9)	2633(10)	2892(4)	7.4(4)
O(1)	3594(8)	5340(7)	2604(4)	3.8(3)
O(2)	3321(7)	3016(7)	1312(3)	3.2(2)
O(3)	5493(7)	5484(7)	1910(3)	3.9(3)
O(4)	1276(7)	3165(8)	1802(4)	3.8(3)
O(5)	2909(9)	5479(8)	1274(4)	4.5(3)
C(1)	3891(10)	3061(10)	2623(5)	2.7(3)
C(2)	4171(11)	3236(10)	3314(5)	3.0(3)
C(3)	4246(11)	2221(10)	3670(5)	3.0(4)
C(4)	4375(14)	2366(12)	4373(5)	4.0(4)
C(5)	3821(15)	3121(14)	4658(6)	5.0(5)
C(6)	3988(19)	3278(16)	5325(7)	6.4(7)
C(7)	4747(22)	2696(18)	5704(6)	7.1(8)
C(8)	5262(23)	1952(18)	5438(6)	7.6(9)
C(9)	5065(17)	1749(14)	4774(6)	5.3(6)
C(10)	4150(13)	951(11)	3364(5)	3.5(4)
C(11)	2869(13)	-328(12)	3292(6)	4.5(4)
C(12)	2780(17)	-1503(13)	3002(7)	5.8(6)
C(13)	3911(23)	-1467(16)	2758(6)	6.3(8)
C(14)	5169(22)	-220(18)	2845(8)	7.1(9)
C(15)	5301(16)	994(13)	3136(7)	5.4(6)
C(21)	2931(14)	2075(14)	765(6)	4.9(5)
C(22)	1566(20)	2404(19)	201(6)	8.6(8)
C(23)	4247(32)	2557(51)	419(12)	18.1(24)
C(24)	2257(38)	646(21)	901(9)	10.5(13)
C(31)	6971(12)	5868(12)	2227(5)	3.8(4)
C(32)	7263(13)	4710(13)	2427(7)	4.9(5)
C(33)	7446(12)	6938(13)	2807(6)	4.2(4)
C(34)	7852(16)	6579(19)	1709(8)	6.8(6)
C(41)	155(11)	2345(13)	2111(6)	4.0(4)
C(42)	207(14)	1061(14)	2333(7)	5.5(5)
C(43)	-1298(15)	1876(18)	1572(9)	6.5(7)
C(44)	215(15)	3247(16)	2691(7)	5.4(6)
C(52)	3952(19)	6561(19)	988(9)	7.7(8)

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1. Fractional Coordinates (X10000) and Isotropic Thermal Parameters (c

C(53)	2971(39)	7014(34)	558(15)	16.3(23)'
C(54)	1736(25)	6701(24)	782(13)	11.0(12)'
C(55)	1487(17)	5500(18)	1121(8)	7.5(7)'
H(1)	3887	2289	2405	2.7
H(2)	4303	4079	3525	3.0
H(5)	3318	3531	4392	5.0
H(6)	3582	3772	5515	6.4
H(7)	4932	2866	6160	7.2
H(8)	5756	1537	5708	7.5
H(9)	5384	1172	4590	5.3
H(11)	2055	-374	3440	4.5
H(12)	1908	-2378	2969	5.8
H(13)	3809	-2281	2534	6.3
H(14)	5988	-173	2698	7.1
H(15)	6208	1854	3189	5.4
H(22)	560	1785	219	8.6
H(22')	1622	2341	-235	8.6
H(22")	1713	3324	315	8.6
H(32)	8284	5081	2646	4.9
H(32')	7050	4084	2054	4.9
H(32")	6657	4214	2707	4.9
H(42)	-583	561	2536	5.5
H(42')	1137	1331	2634	5.5
H(42")	100	464	1971	5.5
H(52)	4350	6174	730	7.7
H(52')	4679	7303	1319	7.7
H(53)	2721	6591	130	15.9
H(53')	3544	8012	579	15.9
H(54)	906	6449	427	11.1
H(54')	1913	7454	1084	11.1
H(55)	1227	5615	1513	7.5
H(55')	736	4671	842	7.5

TABLE II. Anisotropic Thermal Parameters (Angs X 1000)
exp[-19.739(U11hha*a*...+2(U12hka*b*...))]

ATOM	U11	U22	U33	U12	U13	U23
Re(1)	33.1(2)	29.7(2)	33.2(2)	18.2(2)	7.7(1)	4.3(1)
F(21)	113(8)	201(13)	122(9)	114(9)	0(7)	-41(9)
F(22)	425(29)	75(7)	158(13)	82(12)	190(17)	51(8)
F(23)	591(37)	127(11)	109(10)	143(17)	157(16)	-10(8)
F(24)	223(18)	91(9)	264(20)	19(10)	150(17)	-14(10)
F(25)	616(41)	258(19)	75(8)	363(26)	47(14)	35(9)
F(26)	238(15)	101(8)	113(9)	85(9)	22(9)	-15(7)
F(31)	64(4)	79(5)	48(4)	17(4)	13(3)	-5(4)
F(32)	86(6)	56(5)	111(7)	34(4)	18(5)	-11(4)
F(33)	46(4)	101(6)	88(6)	11(4)	-1(4)	-24(5)
F(34)	93(7)	79(6)	103(7)	10(5)	43(5)	39(5)
F(35)	50(5)	135(8)	105(7)	5(5)	39(5)	2(6)
F(36)	96(7)	123(8)	76(6)	9(6)	53(5)	-16(6)
F(41)	66(5)	142(9)	104(7)	64(6)	-1(5)	7(6)
F(42)	48(5)	141(9)	85(6)	25(5)	-3(4)	-40(6)
F(43)	33(4)	138(9)	128(8)	12(5)	18(5)	2(6)
F(44)	76(5)	78(5)	102(6)	49(4)	32(4)	-2(5)
F(45)	73(5)	100(6)	60(4)	51(5)	24(4)	9(4)

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TABLE II. Anisotropic Thermal Parameters (Angs X 1000) (continued)

F(46)	62(5)	118(7)	108(7)	36(5)	53(5)	16(5)
O(1)	61(5)	38(4)	57(5)	33(4)	22(4)	7(3)
O(2)	43(4)	33(4)	37(4)	12(3)	10(3)	-5(3)
O(3)	35(4)	45(4)	53(4)	9(3)	7(3)	16(3)
O(4)	31(4)	55(5)	64(5)	26(4)	8(3)	15(4)
O(5)	67(5)	55(5)	61(5)	40(4)	13(4)	23(4)
C(1)	31(5)	31(5)	49(6)	21(4)	12(4)	2(4)
C(2)	41(6)	34(5)	35(5)	18(5)	4(4)	0(4)
C(3)	44(6)	38(6)	36(5)	23(5)	7(4)	10(4)
C(4)	71(8)	46(6)	41(6)	31(6)	14(5)	3(5)
C(5)	83(9)	63(8)	59(8)	44(7)	24(7)	20(6)
C(6)	120(13)	77(10)	53(8)	51(10)	29(8)	8(7)
C(7)	154(16)	98(12)	42(7)	86(12)	7(9)	3(7)
C(8)	174(18)	107(13)	42(8)	103(13)	13(9)	17(8)
C(9)	107(11)	68(8)	41(7)	61(8)	4(7)	2(6)
C(10)	59(7)	40(6)	53(6)	38(6)	16(5)	11(5)
C(11)	52(7)	37(6)	70(8)	21(6)	-1(6)	-4(6)
C(12)	86(10)	43(7)	75(9)	30(7)	-9(8)	0(6)
C(13)	144(15)	68(10)	52(8)	75(11)	15(9)	8(7)
C(14)	154(16)	81(11)	101(12)	90(12)	79(12)	31(9)
C(15)	81(9)	51(8)	78(9)	32(7)	33(8)	12(7)
C(21)	61(8)	66(8)	47(7)	24(7)	10(6)	-11(6)
C(22)	118(13)	114(14)	33(7)	26(11)	-28(8)	-42(8)
C(23)	118(22)	439(57)	78(17)	97(30)	18(15)	-43(26)
C(24)	237(29)	70(12)	72(12)	50(16)	61(16)	-14(9)
C(31)	38(6)	56(7)	41(6)	16(5)	10(5)	8(5)
C(32)	39(6)	54(7)	90(9)	21(6)	14(6)	2(7)
C(33)	33(6)	53(7)	58(7)	11(5)	3(5)	-1(6)
C(34)	54(9)	85(11)	77(10)	-2(8)	25(8)	-2(9)
C(41)	29(6)	64(8)	54(7)	19(5)	9(5)	8(6)
C(42)	53(7)	59(8)	89(10)	18(6)	30(7)	17(7)
C(43)	44(8)	92(11)	109(13)	31(8)	20(8)	2(10)
C(44)	58(8)	90(11)	75(9)	42(8)	34(7)	22(8)
C(52)	105(12)	114(13)	134(14)	83(11)	68(11)	86(12)
C(53)	308(37)	294(35)	267(32)	278(34)	219(30)	240(30)
C(54)	125(17)	135(18)	203(24)	93(15)	46(17)	98(18)
C(55)	79(10)	103(12)	114(13)	62(10)	-3(9)	44(10)

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TABLE III. Interatomic Distances (A)

Re(1)-O(1)	1.674 (7)	C(1)-C(2)	1.430 (13)
Re(1)-O(2)	1.909 (6)	C(2)-C(3)	1.378 (13)
Re(1)-O(3)	1.968 (7)	C(3)-C(4)	1.473 (14)
Re(1)-O(4)	1.986 (7)	C(3)-C(10)	1.470 (14)
Re(1)-O(5)	2.315 (7)	C(4)-C(5)	1.400 (16)
Re(1)-C(1)	1.917 (10)	C(4)-C(9)	1.389 (16)
F(21)-C(23)	1.264 (32)	C(5)-C(6)	1.392 (17)
F(22)-C(23)	1.403 (50)	C(6)-C(7)	1.383 (20)
F(23)-C(23)	1.366 (28)	C(7)-C(8)	1.338 (20)
F(24)-C(24)	1.177 (29)	C(8)-C(9)	1.383 (17)
F(25)-C(24)	1.535 (35)	C(10)-C(11)	1.385 (15)
F(26)-C(24)	1.315 (18)	C(10)-C(15)	1.371 (16)
F(31)-C(33)	1.331 (13)	C(11)-C(12)	1.367 (18)
F(32)-C(33)	1.348 (14)	C(12)-C(13)	1.372 (21)
F(33)-C(33)	1.326 (12)	C(13)-C(14)	1.351 (23)
F(34)-C(34)	1.306 (20)	C(14)-C(15)	1.384 (18)
F(35)-C(34)	1.333 (17)	C(21)-C(22)	1.842 (22)
F(36)-C(34)	1.295 (17)	C(21)-C(23)	1.574 (28)
F(41)-C(43)	1.344 (18)	C(21)-C(24)	1.443 (24)
F(42)-C(43)	1.291 (17)	C(31)-C(32)	1.484 (17)
F(43)-C(43)	1.362 (17)	C(31)-C(33)	1.510 (16)
F(44)-C(44)	1.344 (16)	C(31)-C(34)	1.568 (17)
F(45)-C(44)	1.331 (15)	C(41)-C(42)	1.511 (18)
F(46)-C(44)	1.348 (14)	C(41)-C(43)	1.551 (18)
O(2)-C(21)	1.391 (13)	C(41)-C(44)	1.536 (18)
O(3)-C(31)	1.402 (12)	C(52)-C(53)	1.496 (26)
O(4)-C(41)	1.404 (13)	C(53)-C(54)	1.366 (28)
O(5)-C(52)	1.433 (16)	C(54)-C(55)	1.451 (23)
O(5)-C(55)	1.466 (16)		

TABLE IV. Intramolecular Angles (Deg)

O(1)-Re(1)-O(2)	174.4 (4)	F(35)-C(34)-C(31)	111 (1)		
O(1)-Re(1)-O(3)	93.7 (3)	F(36)-C(34)-C(31)	111 (1)		
O(1)-Re(1)-O(4)	93.3 (3)	F(41)-C(43)-C(41)	112 (1)		
O(1)-Re(1)-O(5)	87.7 (3)	F(42)-C(43)-C(41)	112 (1)		
O(2)-Re(1)-O(3)	85.7 (3)	F(43)-C(43)-C(41)	111 (1)		
O(2)-Re(1)-O(4)	85.2 (3)	F(44)-C(44)-C(41)	113 (1)		
O(2)-Re(1)-O(5)	86.7 (3)	F(45)-C(44)-C(41)	112 (1)		
O(3)-Re(1)-O(4)	156.5 (3)	F(46)-C(44)-C(41)	112 (1)		
O(3)-Re(1)-O(5)	79.7 (3)	O(2)-C(21)-C(22)	102 (1)		
O(4)-Re(1)-O(5)	78.3 (3)	O(2)-C(21)-C(23)	111 (2)		
O(1)-Re(1)-C(1)	94.7 (4)	O(2)-C(21)-C(24)	112 (1)		
O(2)-Re(1)-C(1)	90.9 (4)	O(3)-C(31)-C(32)	116 (1)		
O(3)-Re(1)-C(1)	102.4 (3)	O(3)-C(31)-C(33)	108 (1)		
O(4)-Re(1)-C(1)	99.3 (3)	O(3)-C(31)-C(34)	103 (1)		
O(5)-Re(1)-C(1)	176.7 (3)	O(4)-C(41)-C(42)	115 (1)		
Re(1)-O(2)-C(21)	170.0 (7)	O(4)-C(41)-C(43)	103 (1)		
Re(1)-O(3)-C(31)	136.3 (6)	O(4)-C(41)-C(44)	110 (1)		
Re(1)-O(4)-C(41)	136.7 (6)	O(5)-C(52)-C(53)	102 (1)		
Re(1)-O(5)-C(52)	125.4 (8)	O(5)-C(55)-C(54)	106 (1)		
Re(1)-O(5)-C(55)	124.2 (8)	C(1)-C(2)-C(3)	121.6 (9)		
C(52)-O(5)-C(55)	109 (1)	C(2)-C(3)-C(4)	120 (1)		
Re(1)-C(1)-C(2)	128.5 (7)	C(2)-C(3)-C(10)	121.7 (9)		
F(21)-C(23)-F(22)	118 (3)	C(4)-C(3)-C(10)	118.1 (9)		
F(21)-C(23)-F(23)	129 (4)	C(3)-C(4)-C(5)	122 (1)		
F(22)-C(23)-F(23)	99 (3)	C(3)-C(4)-C(9)	120 (1)		
F(24)-C(24)-F(25)	117 (2)	C(5)-C(4)-C(9)	118 (1)		
F(24)-C(24)-F(26)	111 (2)	C(4)-C(5)-C(6)	121 (1)		
F(25)-C(24)-F(26)	103 (2)	C(5)-C(6)-C(7)	118 (1)		
F(31)-C(33)-F(32)	105 (1)	C(6)-C(7)-C(8)	121 (1)		
F(31)-C(33)-F(33)	106 (1)	C(7)-C(8)-C(9)	121 (1)		
F(32)-C(33)-F(33)	105 (1)	C(4)-C(9)-C(8)	120 (1)		
F(34)-C(34)-F(35)	105 (1)	C(3)-C(10)-C(11)	120 (1)		
F(34)-C(34)-F(36)	109 (1)	C(3)-C(10)-C(15)	122 (1)		
F(35)-C(34)-F(36)	108 (1)	C(11)-C(10)-C(15)	118 (1)		
F(41)-C(43)-F(42)	108 (1)	C(10)-C(11)-C(12)	120 (1)		
F(41)-C(43)-F(43)	107 (1)	C(11)-C(12)-C(13)	122 (1)		
F(42)-C(43)-F(43)	107 (1)	C(12)-C(13)-C(14)	117 (1)		
F(44)-C(44)-F(45)	106 (1)	C(13)-C(14)-C(15)	122 (1)		
F(44)-C(44)-F(46)	107 (1)	C(10)-C(15)-C(14)	120 (1)		
F(45)-C(44)-F(46)	107 (1)	C(22)-C(21)-C(23)	102 (2)		
F(21)-C(23)-C(21)	106 (2)	C(22)-C(21)-C(24)	111 (2)		
F(22)-C(23)-C(21)	99 (3)	C(23)-C(21)-C(24)	117 (2)		
F(23)-C(23)-C(21)	101 (2)	C(32)-C(31)-C(33)	110 (1)		
F(24)-C(24)-C(21)	114 (2)	C(32)-C(31)-C(34)	110 (1)		
F(25)-C(24)-C(21)	98 (2)	C(33)-C(31)-C(34)	110 (1)		
F(26)-C(24)-C(21)	114 (2)	C(42)-C(41)-C(43)	109 (1)		
F(31)-C(33)-C(31)	111 (1)	C(42)-C(41)-C(44)	109 (1)		
F(32)-C(33)-C(31)	114 (1)	C(43)-C(41)-C(44)	111 (1)		
F(33)-C(33)-C(31)	115 (1)	C(52)-C(53)-C(54)	110 (2)		
F(34)-C(34)-C(31)	113 (1)	C(53)-C(54)-C(55)	106 (2)		

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TABLE V. Intramolecular Non-Bonding Distances (A)

Re(1)...F(21)	4.294	(9)	F(36)...O(2)	3.957	(11)
Re(1)...F(22)	4.112	(12)	F(44)...O(1)	2.981	(10)
Re(1)...F(24)	4.016	(14)	F(44)...O(5)	3.087	(11)
Re(1)...F(25)	4.034	(11)	F(45)...O(1)	2.865	(10)
Re(1)...F(31)	3.570	(7)	F(21)...C(32)	3.670	(17)
Re(1)...F(32)	3.983	(8)	F(22)...C(52)	3.565	(23)
Re(1)...F(36)	4.418	(8)	F(24)...C(41)	3.732	(20)
Re(1)...F(42)	4.468	(8)	F(24)...C(42)	3.267	(21)
Re(1)...F(44)	3.772	(7)	F(25)...C(1)	3.449	(15)
Re(1)...F(45)	3.570	(7)	F(25)...C(14)	3.596	(21)
F(21)...F(25)	2.530	(27)	F(25)...C(15)	3.696	(20)
F(21)...F(26)	3.297	(19)	F(31)...C(1)	3.432	(12)
F(21)...F(36)	3.010	(17)	F(31)...C(2)	3.223	(12)
F(22)...F(36)	3.066	(24)	F(34)...C(52)	3.445	(20)
F(23)...F(24)	3.859	(24)	F(36)...C(23)	3.501	(37)
F(23)...F(25)	3.547	(19)	F(41)...C(55)	3.279	(20)
F(23)...F(26)	2.568	(21)	F(42)...C(22)	3.545	(21)
F(24)...F(42)	3.179	(23)	F(44)...C(55)	3.497	(20)
F(32)...F(34)	2.698	(13)	F(45)...C(1)	3.378	(11)
F(32)...F(35)	3.533	(12)	F(45)...C(2)	3.070	(12)
F(33)...F(34)	3.264	(13)	F(45)...C(5)	3.774	(15)
F(33)...F(35)	2.646	(12)	O(1)...C(33)	3.479	(13)
F(41)...F(44)	2.727	(12)	O(1)...C(44)	3.234	(15)
F(41)...F(46)	3.122	(13)	O(2)...C(32)	3.827	(14)
F(43)...F(44)	3.722	(13)	O(4)...C(22)	3.602	(16)
F(43)...F(46)	2.669	(14)	O(5)...C(22)	3.519	(17)
F(21)...O(3)	3.791	(13)	C(1)...C(15)	3.318	(16)
F(22)...O(3)	3.609	(16)	C(1)...C(32)	3.246	(15)
F(22)...O(5)	3.734	(18)	C(1)...C(42)	3.324	(16)
F(24)...O(4)	3.402	(17)	C(9)...C(11)	3.490	(17)
F(31)...O(1)	2.990	(10)	C(9)...C(15)	3.655	(18)
F(32)...O(1)	3.375	(11)			

TABLE VI. Intermolecular Distances (A)

F(21)...F(41)a	3.090	(13)	F(26)...C(22)c	3.321	(22)
F(22)...F(22)b	2.644	(21)	F(26)...C(54)d	3.292	(22)
F(23)...F(34)b	3.128	(21)	F(31)...C(6)e	3.325	(15)
F(23)...F(42)c	3.247	(18)	F(31)...C(7)e	3.380	(17)
F(26)...F(42)c	3.114	(14)	F(44)...C(32)f	3.364	(13)
F(23)...C(52)b	3.303	(24)	O(1)...C(13)g	3.345	(15)

Symmetry Operation Codes

a	1+X,Y,Z	d	X,-1+Y,Z
b	1-X,1-Y,-Z	e	1-X,1-Y,1-Z
c	-X,-Y,-Z	f	-1+X,Y,Z

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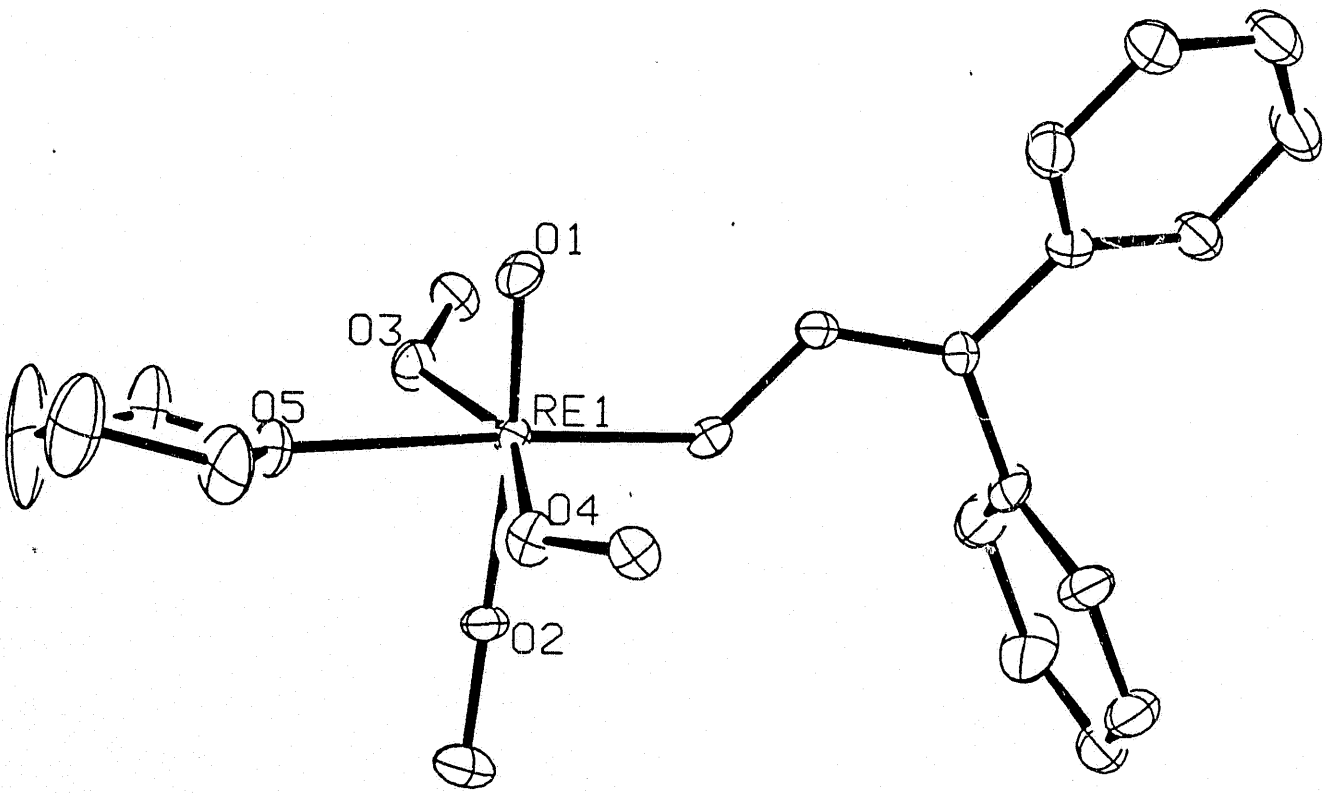

view of oxo-alkylidene - rhenium coordination
Figure 2